

Measured Leak Rate of Ammonia Through an Epoxy/Stainless-Steel Patch

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A handwritten signature in black ink, appearing to read 'D. Robbins', with a long horizontal flourish extending to the right.

Col D. Robbins
SMC/MCWG

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14. ABSTRACT A program has experienced pitting corrosion in anhydrous ammonia-charged loop heat pipes. Areas of each loop heat pipe with increased risk of pitting corrosion were previously identified with X-ray image analysis and patched with a formed stainless-steel cap, bonded with a space-approved epoxy. To assess the lifetime of the patch and sensitivity to manufacturing variation, simulated leaks in circular aluminum discs were patched with several different variants on the same epoxy-stainless materials system. The patches were aged at elevated temperature to accelerate degradation phenomena. No gross failures of the patches were observed in four weeks of testing. No significant difference was observed between the aged bonded material at 353K, 393K, or a freshly prepared patch. Infrared detection of ammonia leaking through the patch suggests that a loss rate of 0.16 ± 0.04 mg of ammonia per year will occur through the simulated leaks with primed patches.					
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1. Introduction

Several anhydrous ammonia-filled loop heat pipes were found to have pitting corrosion in their aluminum 6063 T4 condenser lines. This pitting corrosion could eventually cause small holes to form in the condenser line. This end result was observed on the ground in one commercial program. In order to prevent leakage of ammonia out of these potential holes, 0.020-in.-thick 304 stainless-steel patches were formed, primed (BR 127 black primer), and bonded with epoxy (0.010-in.-thick bond line using EA9323 loaded with glass beads) to the sites where juvenile pits were located after X-ray inspection. The ability of these patched heat pipes to maintain ammonia with a hole in the aluminum condenser was evaluated. The simulated leaks were subject to elevated temperature to simulate on-orbit aging. There was found to be no significant difference in the ammonia leak rate before and after aging. It was determined that the 0.13-mm (5/1000-in.) patched holes would leak 0.16 ± 0.04 mg of ammonia a year, which is expected to bound the orbital leak rate on the upper end.

2. Measuring Sample Storage Tanks by Using Ammonia-Detecting Paint

2.1 Procedure

The test articles were prepared from 0.76-mm-thick aluminum disks (20 mm diameter). The aluminum disks were then “patched” as shown in Table 2.1. The patch was a 6.35-mm-dia piece of either stainless steel or aluminum. The patch was adhered to the simulated pipe aluminum disks by several different epoxy methods as shown in Table 2.1. The epoxy layer was measured as approximately 0.25 mm thick. A 0.13-mm (5-mil) hole was drilled into the center of the aluminum disk. To ensure that the drilled hole was completely through the aluminum, but did not penetrate the patch, the drill depth was set at 0.89 mm. This put the end of the drilled hole about midway through the epoxy layer. Three test articles were disassembled at the conclusion of the test to confirm that the drilled holes were completely through the aluminum, but did not penetrate through the patch material. A picture of a prepared aluminum disk is shown in Figure 2.1.

A 1-L stainless-steel storage vessel was used as the ammonia test tank (Figure 2.2). After preparation, the test articles were placed into the VCR fitting on the storage tank with the hole side towards the tank interior and the patch material on the outside (air side) of the tank. The VCR fitting was then tightened. The storage tank was then placed on a vacuum line and evacuated to less than 0.01 Torr. The valve was closed on the tank, and the evacuated storage tank was then weighed. The storage tank was then placed back on the vacuum line. The valves to the vacuum line were closed, and the ammo-

Tables 2.1. Test Samples Used in the Leak Experiments

Article #	Simulated pipe material	Patch material	Bonder	Primer	Temperature (K)
1	Aluminum	Stainless steel	1	No	393
2	Aluminum	Stainless steel	1	No	393
3	Aluminum	Stainless steel	1	No	353
4	Aluminum	Stainless steel	1	No	353
5	Aluminum	Stainless steel	2	No	393
6	Aluminum	Stainless steel	2	No	353
7	Aluminum	Aluminum	4	No	393
8	Aluminum	Aluminum	4	No	353
9	Aluminum	Stainless steel	1	Yes	353
10	Aluminum	Aluminum	3	No	393
11	Aluminum	Aluminum	3	No	353
12	Aluminum	Aluminum	3	Yes	393
13	Aluminum	Stainless steel	2	Yes	296
14	Aluminum	Aluminum	4	Yes	393
16	Aluminum	Stainless steel	1	Yes	393

1. EA 9323 (with sifted, mil spec cab-o-sil fill, with glass beads)

2. EA 9323 (with glass beads)

3. EA 9323 (with sifted, mil spec cab-o-sil fill, with glass beads)

4. EA 9323 (with glass beads)

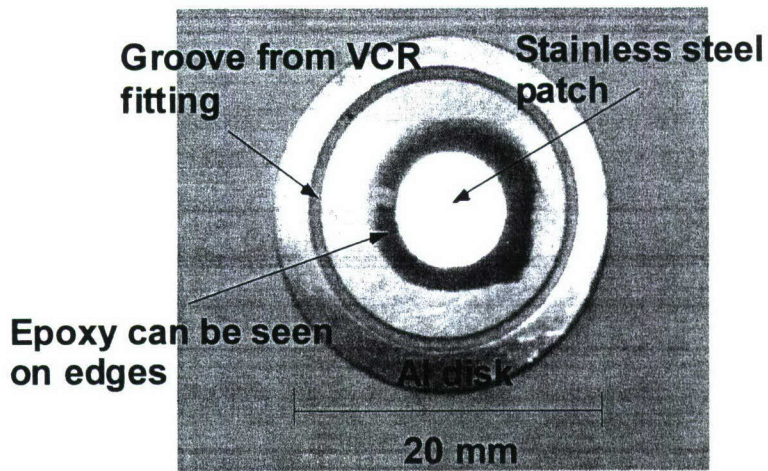


Figure 2.1. A photograph of the aluminum disk used to simulated a patched leaking ammonia heat pipe.

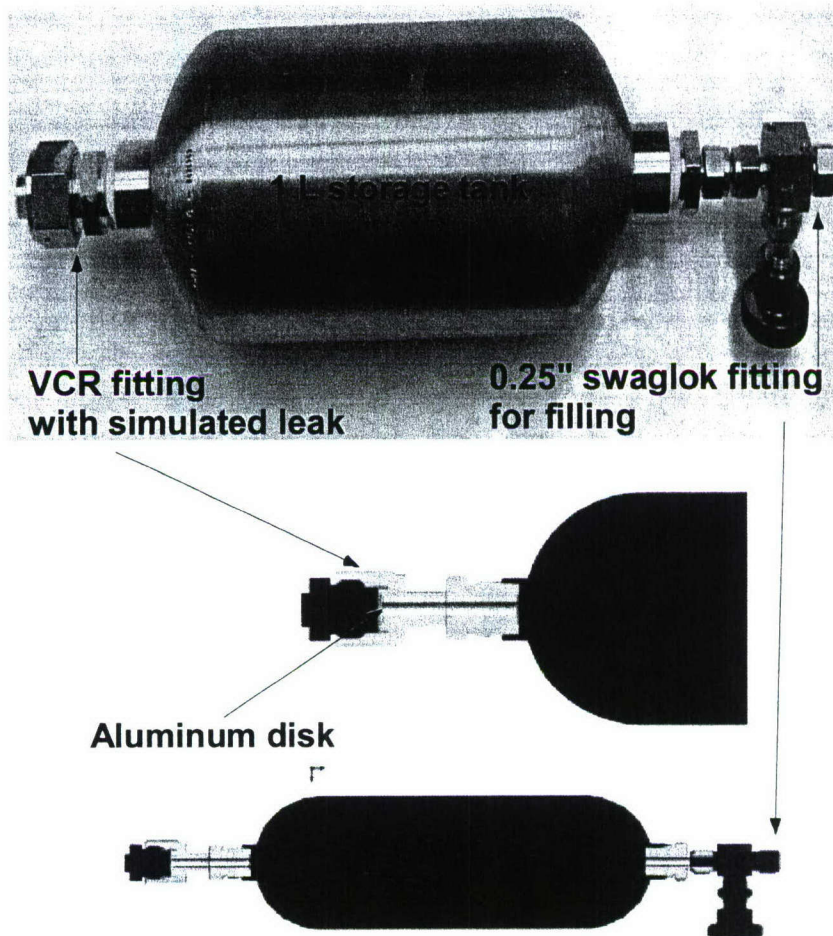


Figure 2.2. Photograph of the ammonia storage tank. The tank volume is 1 L. One end has a valve and a 0.25-in. Swagelok fitting used for filling the tank with ammonia. The other end has a VCR fitting that is used to hold the aluminum disks (used to simulate a patched leaking heat pipe) into place.

nia lecture bottle was then opened. The storage tank was filled with ammonia to about 105 psi. The storage tank and lecture bottle valves were closed, and the remaining ammonia in the line was pumped out. The storage tank was then weighed again. After weighing, the storage tanks were sprayed with ADP 219 (ammonia-detecting paint, a color change indicator for the presence of ammonia). The ammonia detection paint is yellow upon application and will turn blue upon exposure to ammonia. No blue color was observed upon initial application of ADP.

The storage tanks were then placed into two ovens. One oven was kept at 393K, while the other oven was kept at 353K. A test sample was also prepared and stored at lab ambient temperature (296K). An additional test sample that did not have a hole drilled into the aluminum disk (sample number 15) was also prepared and stored at 353K.

2.2 Results

The masses of the filled ammonia tanks are shown in Table 2.2. On average there was 5.0 g of ammonia in each storage tank (105 psi). After the first week, there was no significant measurable loss of ammonia mass. The mass after the second week showed a slight increase in the average weight most likely due to imprecision of the balance. The mass after four weeks showed no significant loss of ammonia mass. No gross leakage of ammonia was detected by weighing the samples stored at either 353K or 393K after four weeks of storage at elevated temperature.

Table 2.2. Measured Mass in Grams of the Storage Tank Samples

Sample number	Initial mass	Filled mass	Δ mass after 1 week	Δ mass after 2 weeks	Δ mass after 4 weeks
1	3113.5	3119.4	-0.1	0.1	0.0
2	3211.8	3217	-0.1	0.1	0.1
3	3163.2	3168.4	0	0.2	0.1
4	3191.9	3196.9	-0.2	0	-0.01
5	3164.5	3169.9	0.2	0.4	-0.1
6	3099.2	3104.3	-0.1	0.3	0.1
7	3156.6	3161.7	0.1	0.3	0.0
8	3191.5	3196.5	0.0	0.0	-0.1
9	3184.9	3189.3	0.2	0.6	0.2
10	3147.2	3152.4	0.0	0.0	0.0
11	3104.8	3109.5	0.1	0.3	-0.1
12	3111.2	3115.5	0.0	0.3	-0.2
14	3133.7	3138.5	0.0	0.3	-0.1
16	3147.8	3152.8	0.1	0.0	0.0
average	3151.557	3156.579	0.013	0.200	-0.007

After two weeks of storage, all samples had shown a change in the color of the ADP from yellow to blue when the test articles were cooled to lab ambient for weighing, indicating detection of ammonia. However, at the accelerated aging temperature, the ADP was still yellow. This observation led to the hypothesis that ADP color change is temperature dependent. ADP was applied to an aluminum foil sample. This sample was exposed to ammonia to turn the ADP blue. The sample was then placed into an oven at 353K for a few hours. The sample returned to a yellow color at this temperature. After removal, the sample returned to a blue color (see Figure 2.3). A similar sample was made but not exposed to ammonia. It did not turn blue after heating to 353K and returning to room temperature. Thus, the change in color of the tank samples indicated possible exposure to ammonia, though at a level less than could be detected by mass loss from the tanks. Further investigation determined that two of the storage tanks (test articles 4 and 16) were responsible for the ADP color change. One leaked at the VCR fitting, and another leaked at a pipe thread fitting. These two leaks were easily detected by ADP. One leaking tank was located in each oven. These two leaks likely caused all the other test samples in the ovens to turn blue. It is reasonable that in the enclosed oven,

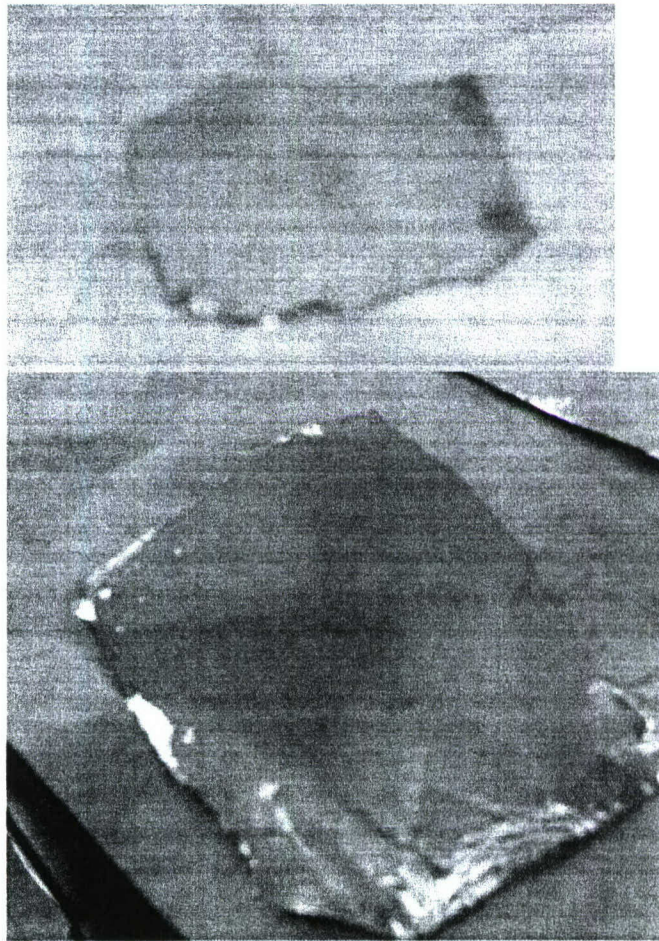


Figure 2.3. Aluminum foil sample that has been painted with ADP and then exposed to ammonia gas. The top picture is directly after removal from a 353K oven. The bottom picture is after returning the foil to room temperature.

over the course of two weeks, a background of ammonia would have built up. Aluminum foil samples placed in the ovens for two weeks also turned blue, consistent with this conclusion. The test blank (test article 15 without a hole) in a separate oven at 353K did not turn blue, indicating that exposure to room air alone will not change the ADP color. Also, several test samples with ADP painted on aluminum foil and left out in lab air over the two-week period did not turn blue. At the end of four weeks, the storage tanks were removed from the oven, and ADP was reapplied. There was no observed color change, indicating that no gross leakage of ammonia from the patches has formed from four weeks of aging.

Figure 2.4 shows the color of the ADP of a 353K-exposed sample before and after two weeks of heating. At the two-week point, the ADP paint was removed from the aluminum disks with compressed air. The aluminum disks were then repainted with ADP, but after two days, no color change occurred.

The epoxy showed a color change after heating. The control sample remained off-white throughout the four-week study. The epoxy on the samples at 353K for four weeks changed to a tan color. The epoxy on the samples that were placed at 393K for four weeks turned a dark brown. The color change was not correlated with any observed increase in the leak rate from the samples. The color change of the epoxy is shown in Figure 2.5.

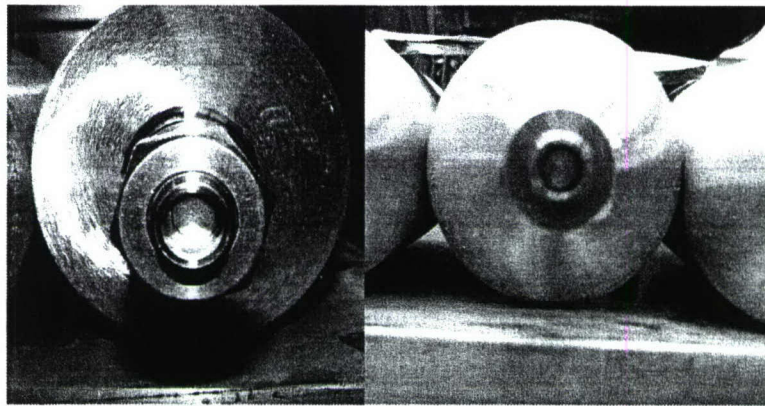


Figure 2.4. A storage tank filled with ammonia before testing for 2 weeks at 353 K. At left the ADP is seen to be yellow indicating no exposure to ammonia. At right the ADP has turned blue indicating exposure to ammonia.

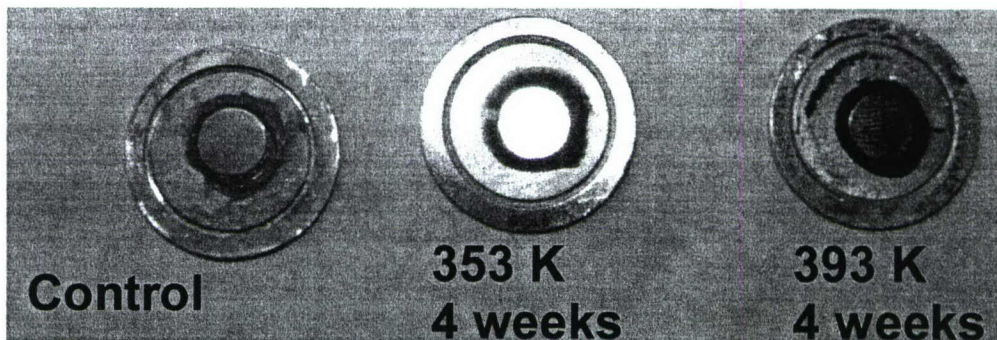


Figure 2.5. Photograph of aluminum disks after testing. The left is the control disk that was not heated, the middle sample was heated to 353K for 4 weeks, and the right sample was heated to 393K for 4 weeks.

2.3 Conclusion

Mass loss measurements of the samples showed no significant mass loss over a four-week period, indicating no catastrophic failure of any patch. The ADP did turn blue over the first two-week period for every test article. However, it was determined that these were false positives caused by leaks in two storage tanks at connections unrelated to the simulated patched holes. No test article turned blue over the course of a few days after the storage tanks were returned to room temperature and unrelated leaks were repaired. After four weeks, the color change indicator paint showed no significant leak had formed in the patch material. Thus, the ADP test and the mass measurements were in agreement that no catastrophic failure of the patch material occurred.

3. Infrared Quantification of the Ammonia Leak Rate from Primed and Unprimed Stainless-Steel Samples

3.1 Procedure

The high-resolution IR absorption of ammonia in the gas phase was used to follow the leak rate of ammonia through the patch over a period of days. A diagram of the IR laser setup used to measure the ammonia storage tank samples is shown in Figures 3.1 and 3.2. The ammonia concentration was monitored using a tunable diode infrared laser. The IR absorption of ammonia was observed at 6691.836 cm^{-1} , the R(6) of the $\nu_1 + \nu_3$ band.¹ The IR probe was split into two beams by a CaF_2 flat. The first beam was directed through a short-focus lens onto an infrared detector (reference). The second beam was directed into a Herriott-type multipass cell.² The cylindrical cell (volume 142 cm^3) was 10 cm long with a 2 cm radius, and has anti-reflective-coated windows. The multipass cell is usually set up to perform 63 passes. The total path length of the infrared laser in the multipass cell is $\sim 630\text{ cm}$. After exiting the cell, the second beam was directed through a short-focus lens onto an infrared detector (signal). The laser frequency was then tuned over an ammonia absorption feature. In order to reduce laser noise in the ammonia signal, the absorption was monitored as the difference between the balanced signal and reference detectors.

The IR cross section of the ammonia transition used to observe ammonia formation was obtained in order to convert absorption into a concentration. Lundsberg-Nelsen et al. have previously measured

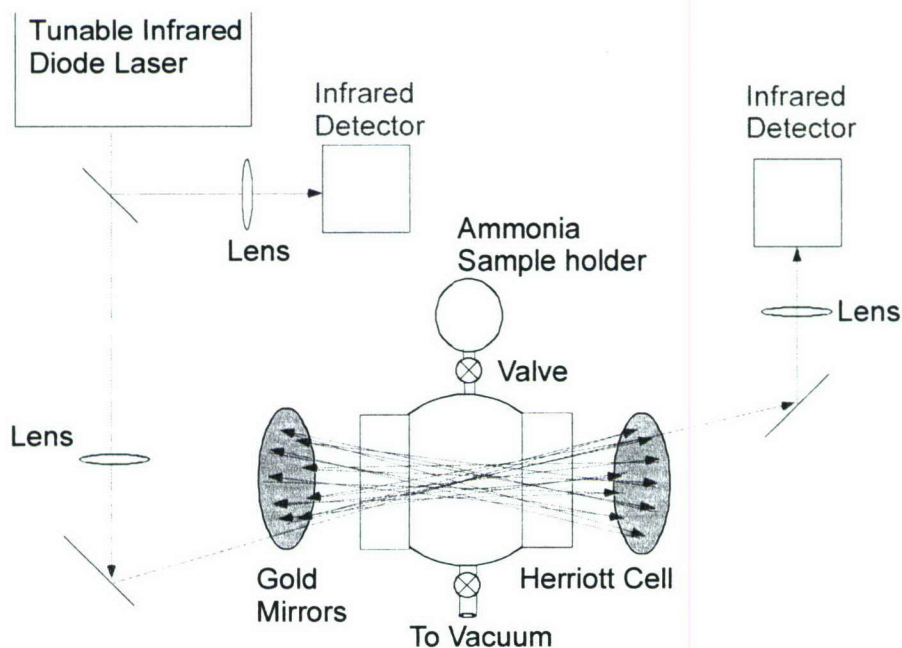


Figure 3.1. The IR laser apparatus setup.

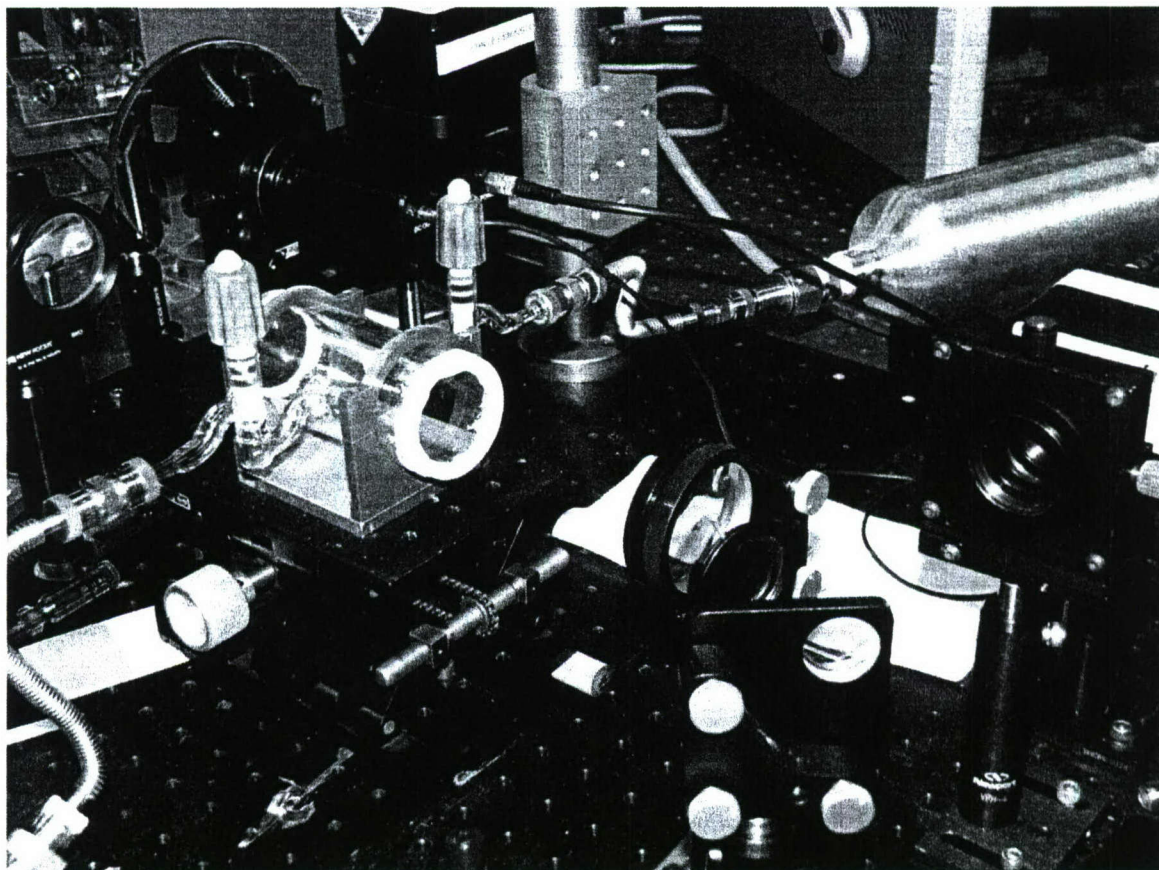


Figure 3.2. Photograph of the IR laser absorption experimental setup.

the cross section to be $7.28 \times 10^{-20} \text{ molec}^{-1} \text{ cm}^2$ at 294.15K. Figure 3.3 shows the observed absorption of the ammonia transition at different ammonia concentrations using a 30-cm path length and a 151-cm path length. The cross section can be obtained from the slope of a linear fit to the data in Figure 3.3 using the Beer-Lambert law;

$$\sigma_{\text{IR}} = \frac{\text{Log}\left[\frac{I}{I_0}\right]}{L \times [\text{NH}_3]}, \quad (3.1)$$

where I is laser intensity and L is the path length. The slope yields a cross section of $\sigma_{\text{IR}} = 7.6 (\pm 0.4) \times 10^{-20} \pm 0.4 \text{ cm}^2$. This is in good agreement with the previous measurement.

3.2 Results

Figure 3.4 shows the infrared spectrum of ammonia with 0.9 Torr of ammonia in the test cell (black). Also shown is the infrared spectrum of ammonia for test sample tank 16 after leaking into the test apparatus for 67.25 h. Equation (3.1) was used to determine the amount of ammonia present from the ammonia infrared signal. The signal in Figure 3.4 corresponds to an increase in ammonia of $3.19 \times 10^{14} \text{ molec cm}^{-3}$ in the cell.

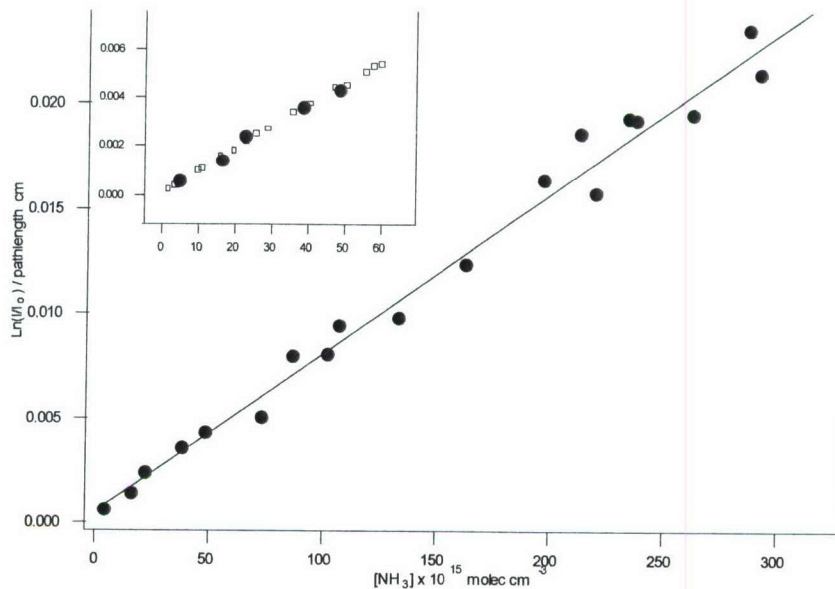


Figure 3.3. The absorbance of the IR laser divided by the path length vs. the ammonia concentration. The slope corresponds to an IR cross-section of $\sigma_{\text{IR}} = 7.6(\pm 0.4) \times 10^{-20} \pm 0.4 \text{ cm}^2$.

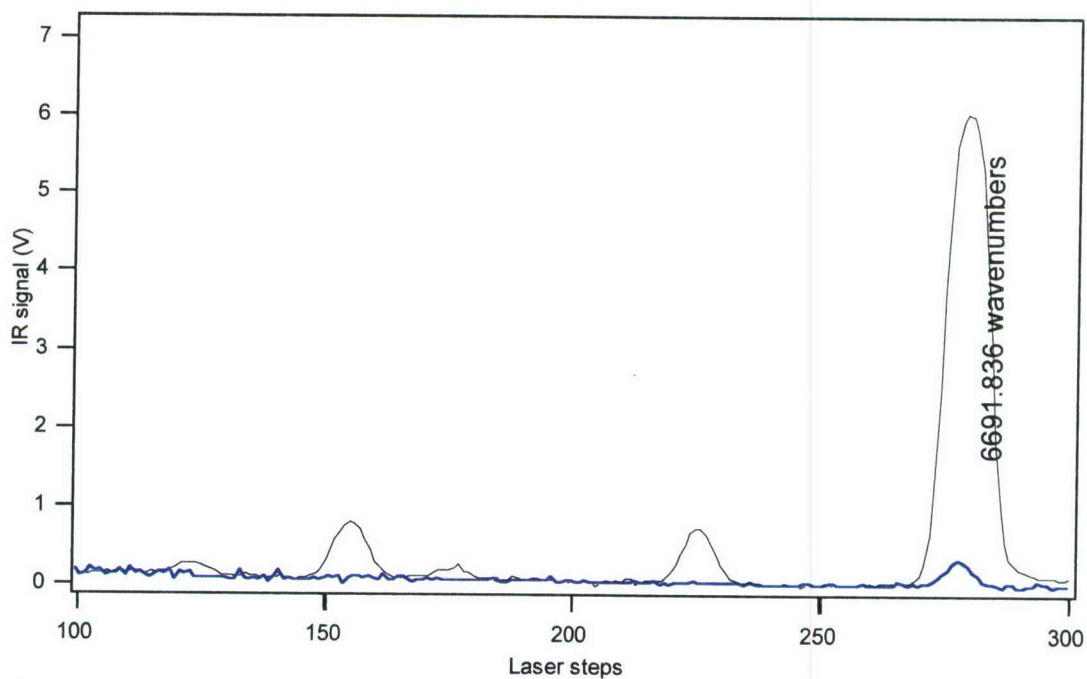


Figure 3.4. The IR absorption of ammonia spectrum around 6691.836 cm^{-1} . The black spectrum was a test spectrum done with 0.9 Torr of ammonia in the cell. The blue spectrum was taken after sample tank 16 leaked into the cell for 67.25 hrs.

The ADP was observed to outgas off the surface of the storage tanks. The ADP outgassing product has an absorption peak near the ammonia peak used to monitor the experiment (Figure 3.5). The peaks are far enough from each other (in wavelength) so as not to cause interference. Figure 3.6

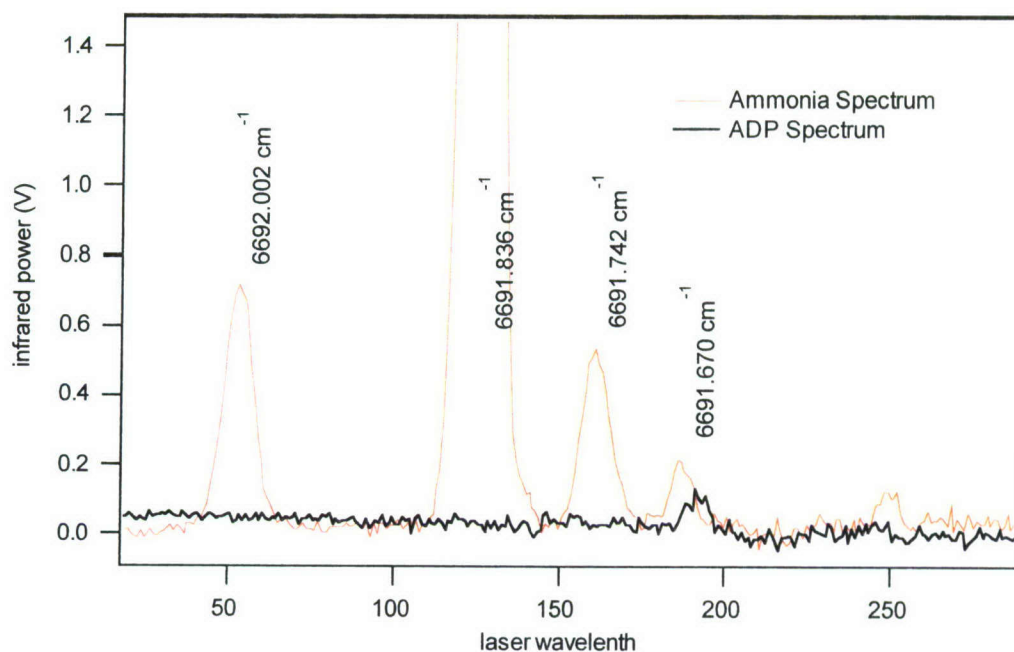


Figure 3.5. The IR absorption of the ADP spectrum around 6691.6 cm^{-1} . The red spectrum was taken with ammonia in the test cell. The black spectrum was taken of the out gassing from an aluminum foil sample that had been sprayed with ADP and not exposed to ammonia.

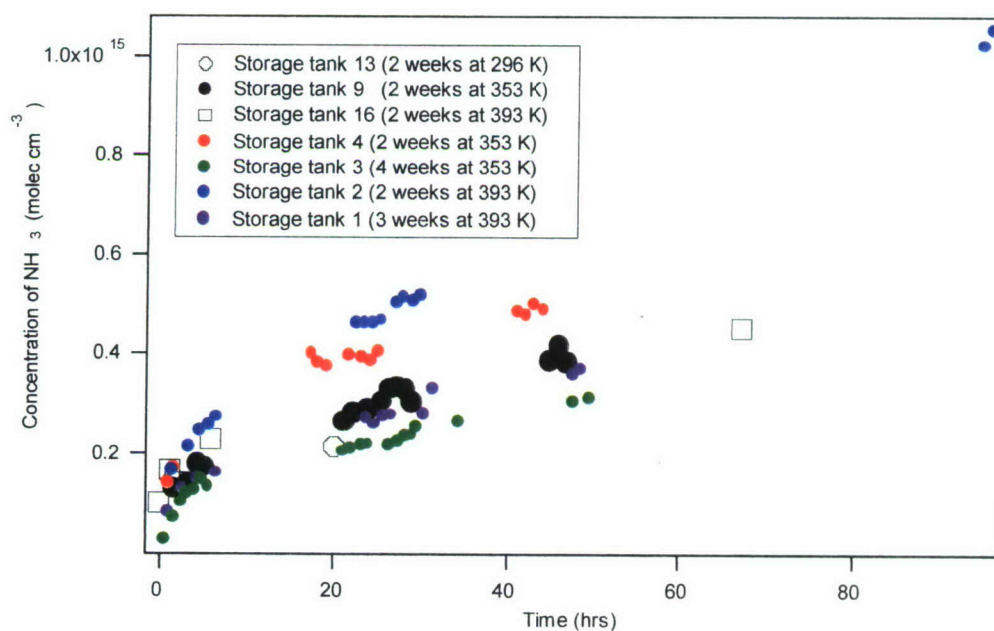


Figure 3.6. The observed increase in ammonia in the test cell for 7 different test samples. The (\circ) sample was kept at room temperature, the (\bullet) sample was stored for 2 weeks at 353K and the (\square) sample was stored for 2 weeks at 393K, the red (\bullet) sample was stored for 2 weeks at 353K, the green (\bullet) sample was stored for 4 weeks at 353K, the blue (\bullet) sample was stored for 2 weeks at 393K and the violet (\bullet) sample was stored for 3 weeks at 393K. The leak rates of ammonia for the three samples were all observed to be similar.

shows the increase in ammonia with time for test tanks 1, 2, 3, 4, 9, 13 and 16. After the initial first day of outgassing from the walls of the test cell, the amount of ammonia increased linearly with time over several days, corresponding to leak rates shown in Table 3.1. There is no significant difference in the leak rate between the samples with (9,13 and 16) and without (1, 2, 3, 4) the primer. An extra week of storage at 393K (1) or two weeks at 353K (3) had no significant effect on the measured ammonia leak rate.

The average leak rate for the ammonia was $5.0 \pm 1. \times 10^{-7}$ sccm. Using this data, along with the exposed area of the epoxy, the path length through the epoxy, and the pressure of ammonia in the storage tank, we can calculate the permeation rate through the simulated patch. The ammonia permeation rate through the patch material was measured to be $5. \pm 1.3 \times 10^{-6}$ sccs mm atm cm² at room temperature. This permeation rate was found to be lower than the industry standard number for the epoxy material. The lower measured permeation rate in these experiments may be due to the use of impermeable glass beads in the epoxy.

Table 3.1. Leak Rate of Sample Tanks as Measured by IR Absorption

Sample tank	Leak rate (molec cm ⁻³ h ⁻¹)	Leak rate SCCM
13 (296 K)	3.39×10^{12}	3.02×10^{-7}
9 (353 K)	5.85×10^{12}	5.22×10^{-7}
16 (393 K)	4.63×10^{12}	4.13×10^{-7}
1 (393 K) ^a	5.58×10^{12}	4.98×10^{-7}
2 (393 K)	8.20×10^{12}	7.32×10^{-7}
3 (353 K) ^b	3.99×10^{12}	3.56×10^{-7}
4 (353 K)	7.17×10^{12}	6.40×10^{-7}

a. Stored for 3 weeks at 393 K

b. Stored for 4 weeks at 353 K

3.3 Conclusion

As seen in Figure 3.6, the leak rates of the seven samples were all very similar. There was no significant effect of storage in an ammonia environment for 2, 3, or 4 weeks at either 353K or 393K on the ability of the patches to prevent ammonia from leaking through the simulated hole. The average leak rate in Table 2.2 corresponds to a loss of 0.16 ± 0.04 mg of ammonia annually from the simulated primed patched-hole samples. The patch sample without the primer had a slightly larger leak rate, which corresponds to a loss of 0.19 ± 0.04 mg ammonia annually.

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1. Lundsberg-Nelsen, L.; Hegelund, F.; Nicolaisen, F. M. *J. Mol. Spectrosc.* **1993**, 162, 230.
2. Herriott, D.; Kogelnik, H.; Kompfner R. *Appl. Opt.* **1964**, 3, 523.

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